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TECHNICAL STUDIES SUPPORTING THE MINING WASTE REGULATORY DETERMINATION

FINAL REPORT

Contract No. 68-01-7053
Work Assignment 42
Tasks 1 and 2

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1. INTRODUCTION

EPA is concerned with the applicability of the EP (Extraction Procedure)-Toxicity Test when evaluating the RCRA hazardous characteristics of mining and smelting wastes. The lead and cadmium values generated by the EP-Toxicity Test for mining and smelting wastes are of particular concern. Since approximately 11 million metric tons of mine and mill wastes and more than a million tons of smelting wastes generated annually, exceed EP-Toxicity maximum contaminant limits, it is important to understand whether the EP-Toxicity Test for these sample types is both accurate and realistic. Two laboratory tasks were undertaken:

Task 1: Assess the validity of As, Pb, Cd, Ba, Ag, and Cr concentrations in EP-Toxicity leachates.

Task 2: Compare the extraction efficiency of four leachate techniques.

To accomplish Task 1, the samples were processed, in duplicate, according to three different preparation procedures (EP-Toxicity Test and two acid digestion procedures). After analysis by ICP, the element data generated by the acid digestions were compared to the amount of element extracted by the EP-Toxicity Test. This comparison of the potentially extractable element content in the acid digestates with the observed EP levels was used to confirmed the validity of the EP-Toxicity Test for these sample types.

In Task 2, four extraction procedures were performed on the mining waste samples:

1. EP-Toxicity Test;
2. EP-Toxicity Test without pH adjustment;
3. ASTM Extraction Procedure; and
4. Synthetic Rainwater Leach.

The data generated by the four extractions were compared to determine any differences between methods as well as to provide a better understanding of factors influencing element release from these sample types.

2. CONCLUSIONS AND RECOMMENDATIONS

2.1 Task 1 Conclusions

In Task 1, the validity of As, Ba, Cd, Cr, Pb, and Ag concentrations in the EP-Toxicity Test leachates for mining and smelting wastes was tested. Conclusions that can be made, based on this limited study of four samples, are as follows:

- Total metals analysis data, were found to be greater than observed EP-extraction data for these mining waste samples. Concern that the EP-Toxicity Test generates data for mining and smelting wastes that are higher than the "total" metal content, appears to be unfounded.

- Duplicate sample precision was generally good (RSD <20 percent). Errors due to nonhomogeneous samples or imprecision in the preparation or analytical techniques were not a factor in data evaluation.

- Accuracy of the ICP method was measured by bench spiking all samples. Spike recoveries were generally in acceptable ranges (80 to 120 percent), indicating that the ICP method did not significantly bias the results.

2.2 Task 2 Conclusions

In Task 2, the EP-Toxicity Test was compared with three other extraction procedures to determine if the EP-Toxicity Test is an appropriate method for determining the toxicity of mining and smelting wastes.

The conclusions are as follows:

- The extraction pH, and not the affinity of acetic acid (as used in the EP-Toxicity Test), appears to be the dominant factor in regulating extracted metal levels. Generally, the lower the extract pH value (as in the Synthetic Rainwater Leach and EP-Toxicity Test) the greater the observed metal concentrations. Conversely when the extract pH values were higher (as in the ASTM and EP-Toxicity Test without pH control), the observed metals levels were lower. These are general observations and were not rigorously tested.

- Where pH control was not used, as in the ASTM and EP-Toxicity Test without pH control, the extraction solid/liquid ratio probably controls the leachate metal levels.

- The Synthetic Rainwater Leach was a rapid technique. Duplicate results, however, showed poorer precision than other extraction methods. Greater quantities of metals were leached out by the Synthetic Rainwater Leach than any other procedure. This is understandable, since the pH of the extraction fluid (3.98) was the lowest used in the study.

2.3 Recommendations

- To accurately assess pH control of metal release, a rigorous statistical study employing several different acids and pH levels would be necessary.

- The Synthetic Rainwater Leach may be a viable alternative to the EP-Toxicity Test, but a more thorough study would be necessary to define experimental parameters as well as to determine method precision.

3. DESCRIPTION OF STUDY

3.1 Study Design

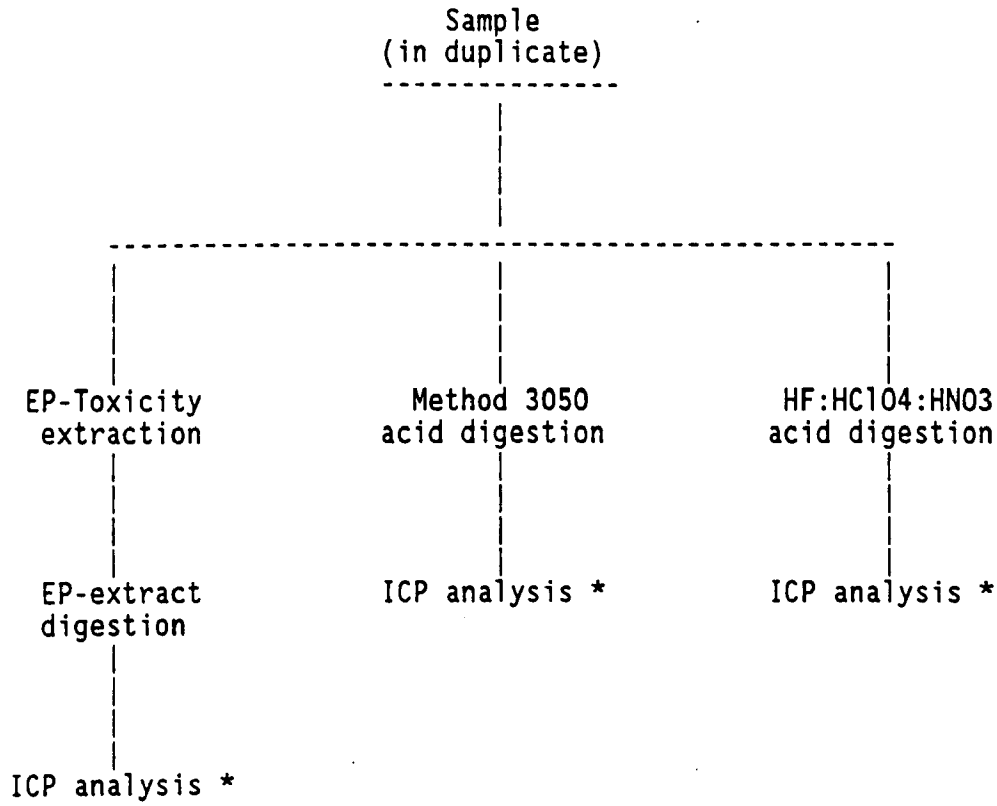
In Task 1, three preparation procedures were carried out on the four samples selected for use in the study. A fifth sample provided by EPA (an EP-QC sludge) was also included. Figure 3-1 provides a schematic description of the preparation and analysis procedures. All samples in this task were run in duplicate to determine method precision. Also, bench spikes of the six study elements (As, Ba, Cr, Cd, Pb, and Ag) were performed on all samples to investigate ICP accuracy in these matrices.

In Task 2, the EP results on the samples from Task 1 were compared with the results from three additional extraction procedures carried out on the same samples. A schematic description of this study is provided in Figure 3-2. Again, all sample extracts were bench spiked and reanalyzed to evaluate the accuracy of the ICP method in these sample matrixes.

3.2 Materials/Instrumentation

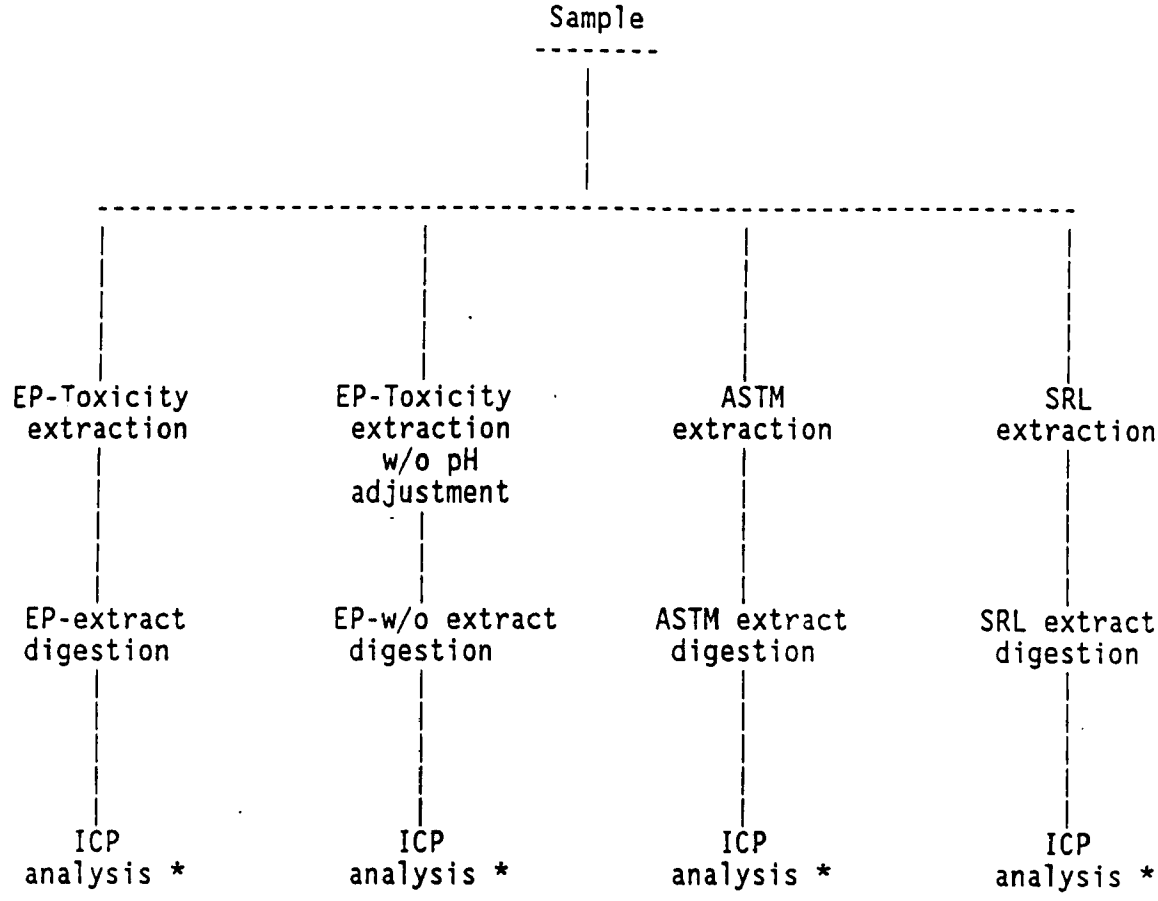
3.2.1 Samples

There were five samples used in this study. Four of the samples were supplied by Bob Hoy of PEI Associates in Cincinnati, Ohio. The fifth sample was supplied by Florence Richardson, Quality Assurance Officer, of the EPA's Office of Solid Waste. A list of the samples follows:



* All samples were bench spiked (after digestion) and reanalyzed.

Figure 3-1 Task 1 Extraction and Analysis Scheme



* All samples were bench spiked (after digestion) and reanalyzed.

Figure 3-2 Task 2 Extraction and Analysis Scheme

Lab Sample No.	Field Sample No.	PEI Sample Description
2023	DR 089	Sn Smelter Slag
2024	DR 950	Pb/Zn Smelter Slag
2025	DR 713	Cu Smelter Slag
2028	DQ 231	Pb Smelter Slag
----	EPA Preaward 1	Solid Waste

3.2.2 Materials

- Acids:
  - Baker Reagent Grade HF
  - Baker Instra Analyzed HNO_3
  - Baker Ultrex $HClO_4$
  - Fisher Glacial Acetic Acid
  - Fisher Reagent Grade HCl
- Mallinckrodt Reagent Grade 30 percent H_2O_2
- NBS Aqueous Spectrometric Standard Reference Materials
- Diamonite mortar and pestle
- Dynalon PTFE Teflon beakers
- Pyrex glassware (acid cleaned)
- Nalgene pressure filtration apparatus

3.2.3 Instrumentation

- Orion Research pH meter model 501
- Eberbach horizontal extraction shaker
- Sybron/Thermolyne 30400 furnace
- Mettler 440 analytical balance
- Rotary 6 place EP-extraction box
- Labconco Micro Kjeldahl digestion rig
- Jarrell-Ash 1150 Inductively Coupled Argon Plasma Spectrometer

3.3 Methods

3.3.1 Sample Preparation

There were two solid digestion procedures and four solid/liquid extraction procedures used in Tasks 1 and 2. Additionally, a liquid

digestion procedure was required following the solid/liquid extraction. Outlines of these methods follow with method references provided at the end of the section.

HF:$HClO_4$:HNO_3 Sample Digestion[1] - ("Total")

- Sample is pulverized using a mortar and pestle;
- Sample is dried at 60^0 C;
- A 0.5 gram sample is weighed into a Teflon beaker;
- Five mls of 48 percent HF are added;
- Sample is brought to dryness on a steambath;
- The residue is transferred to a 100 ml Kjeldahl flask;
- Ten ml of a 5:3 HNO_3:$HClO_4$ solution are added;
- Sample is heated in a Kjeldahl digestion rig until the evolution of $HClO_4$ fumes;
- Five ml of HCl are added and the sample is heated for one hour; and
- The sample is cooled, filtered (if necessary), and brought to a final volume of 100 ml.

Acid Digestion of Sludges[2] - (3050)

- One gram of dried sample is weighed into a 150 ml beaker;
- Ten mls of 1:1 HNO_3 are added and the sample is refluxed for ten minutes;
- Five mls of HNO_3 are added and the sample is refluxed for another 30 minutes;
- The sample is cooled and two mls of DI and three mls of H_2O_2 are added;
- The sample is heated to promote the peroxide reaction;
- Additional H_2O_2 is added as needed (10 mls maximum);
- The sample is cooled; five mls of 1:1 HCl and ten mls of DI are added; the beaker is warmed; and
- The sample is filtered (Whatman No. 41) and brought up to a final volume of 100 mls.

EP-Toxicity Test[2] - (EP)

- A 100 g of sample is weighed out and separated into its component phases by pressure filtration.
- The filtrate is stored; the solid portion is placed in an extraction bottle and 16 times its weight in DI is added.
- The sample is extracted over a period of 24 hours in a rotary extractor; the pH of the extraction fluid is controlled to a pH of 5.0 with 0.5 N acetic acid. No more than four mls of extraction fluid per gram of sample is added to the sample.

- At the conclusion of the 24 hour period the final volume is adjusted to 20 times the sample weight.
- The solid and liquid phases are separated by pressure filtration and the filtrate is combined with the initial liquid phase as the EP-leachate.
- The sample is now ready for a 6010 digestion and analysis.

EP-Toxicity Test Without pH Adjustment[2] - (EP-w/o)

- Same procedure as in the previous EP-Toxicity Test without the addition of the acetic acid for pH adjustment.

ASTM Extraction Procedure[3] - (ASTM)

- 350 grams of sample are weighed into an extraction bottle.
- DI is added at a ratio of 4:1 (1400 mls).
- The container is closed and inverted at a rate of 25 times/minute for three minutes.
- The sample is placed on a horizontal extraction shaker and extracted for 48 hours at 60 to 70 cycles/minute.
- The solid and liquid phases are separated by pressure filtration.
- The sample is now ready for 6010 digestion and analysis.

Synthetic Rainwater Leach - (SRL)

- Ten grams of sample are weighed out into a plastic container.
- Two hundred mls of Synthetic Rainwater (similar to NBS SRM 2694-I) are added. This solution can be purchased or prepared as specified in reference 4. The chemicals used to make up this solution are listed in Table 3-1.
- The container is closed and placed on a wrist action shaker for one hour.
- The sample is centrifuged and filtered.
- The extract is now ready for 6010 digestion and analysis.

Method 6010 ICP Digestion for Aqueous Samples, par. 7.3[2]

- Fifty mls of sample are transferred to a beaker.
- Three mls of HNO_3 are added and the sample is evaporated to near dryness.
- The sample is cooled and an additional three mls of HNO_3 are added.
- The sample are refluxed for one hour.

Table 3-1 Synthetic Rainwater Contents

Compound	Concentration in SRL * (in mg salt/liter)
$NaNO_3$	0.491 mg
KNO_3	0.130 mg
$CaCl_2 \times 2H_2O$	0.057 mg
$MgSO_4 \times 7H_2O$	0.205 mg
NH_4Cl	0.300 mg
H_2SO_4	0.025 mmol
HNO_3	0.050 mmol
NaF	0.117 mg

* Based on NBS SRM 2694-I. Koch, W.F., Marimenko, G., and Paule, R.C. 1986 (in publication). Development of a standard reference material for rainwater analysis. J.Res. NBS (draft) 91(1):____.

- Five mls of 1:1 HCl and ten mls of DI are added and the beaker is warmed.
- The sample is cooled, filtered if necessary, and brought up to a final volume of 50 mls.

References:

1. Procedures for handling and chemical analysis of sediment and water samples. May 1981. Technical Report EPA/COE, CE-81-1.

2. Test methods for evaluating solid wastes, physical/chemical methods. SW 846, U.S. Environmental Protection Agency, 1982.

3. Shake Extraction of Solid Waste with Water. ASTM Method D 3987-81.

4. Development of a standard reference material for rainwater analysis. Koch, W.F., et al. 1986. Journal of Research of the National Bureau of Standards. Vol. 91, No. 1 (in publication).

3.3.2 ICP Analytical Procedures

Samples are run on a direct reading Jarrell-Ash 1150 Inductively Coupled Argon Plasma Spectrometer (ICP). The instrument is outfitted with a sophisticated, computer-controlled (DEC PDP 11-23), background correction and data management system. The spectrometer is currently configured for simultaneous analysis of 32 elements.

A two-point standard calibration process is followed employing a 3 percent HNO_3 blank solution and a standard solution at one or ten mg/L (depending on the element). Computer-fitted linear regression curves are calculated for comparison with unknowns.

Samples from each of the six preparation schemes were run in the following manner. The sample was run straight (1X) and then diluted (if necessary) and rerun. A dilution on a sample was deemed necessary if the concentration of any of the six required elements (As, Ba, Cd, Cr, Pb, Ag) were outside their linear range. Dilutions were also performed

when high concentrations of other elements were suspected to interfere in element quantification. Additionally, samples were spiked with each with the six elements and were reanalyzed to evaluate the accuracy of the ICP method on those sample types. Reporting limits for the six elements can be found in Table 3-2. These limits changed for some samples when dilution was necessary.

3.3.3 Program Quality Assurance Measures

(1) Task 1. Laboratory quality assurance measures included preparation blanks, duplicates, and spikes, the frequency of which is reported in Table 3-3. To obtain the best information on biases due to sample heterogeneity and preparation/analysis errors, every sample in the three preparation procedures ("Total", 3050, and EP) was duplicated. As mentioned previously, all samples were bench spiked to investigate the accuracy of the ICP method in the study matrices. To check for instrumental drift and standard accuracy, EPA reference vials were analyzed prior to each analysis and at a frequency of five percent. If concentrations of the six elements fell outside of control limits for the EPA solutions, the analysis was terminated, the problem corrected, and any samples analyzed up to the failed check sample were reanalyzed.

To assist in verifying the accuracy of the EP-Toxicity Test a QC sludge sample (EPA Preaward Sample #1) provided by EPA was analyzed. The sample was prepared and analyzed according to all three procedures.

(2) Task 2. QC frequency for Task 2 is also provided in Table 3-3. Only one duplicate was analyzed in the EP-w/o, ASTM, and SRL preparation

TABLE 3-2 ANALYTICAL METHODS/REPORTING LIMITS

Element	Method	Reporting Limit mg/l
Arsenic	6010 ICP	0.05
Barium	6010 ICP	0.003
Cadmium	6010 ICP	0.004
Chromium	6010 ICP	0.004
Lead	6010 ICP	0.05
Silver	6010 ICP	0.01

Table 3-3 Frequency of Quality Control Measures

Preparation procedure	Task	Number of QC Measurements: Preparation blanks	Duplicates	Matrix * spikes	Bench ** spikes
"3050"	1	1	All samples	1	All samples
"Total"	1	1	All samples	1	All samples
EP	1,2	1	All samples	0	All samples
EP w/o	2	1	1	0	All samples
ASTM	2	1	1	0	All samples
SRL	2	1	1	0	All samples

* Matrix Spikes - Spikes made into the sample prior to and carried through preparation procedure.

** Bench Spikes - Spikes made after sample preparation, but prior to instrumental analysis.

schemes; however, the same frequency of blanks and bench spikes were analyzed as in Task 1.

4. RESULTS AND DISCUSSION

4.1 Task 1 - Validation of Element Measurements in EP

4.1.1 Task 1 Results and Quality Control Data

Four smelter slag samples and an EPA QC-sludge sample were used in the study. The slag samples were previously homogenized by PEI and contained little water. The EPA material was a mud and contained more than 50 percent water. A physical description of these samples and their percentage of solid content are provided in Table 4-1.

The analytical data for the "Total", 3050, and EP extracts are displayed in Table 4-2. In Appendix 1, the final reported value of each sample is provided, along with the raw unadjusted concentration in the digestate, dilution factors, and bench spike data.

Preparation blank values were insignificant compared to sample data and in almost all cases were below detectable limits. Duplicate RPD values were <20 percent, except for some elements when they were near the detection limit, and in two cases for Ba in the EP-extract. Matrix spike recoveries were in the acceptable range of 80 to 120 percent in the two acid digestion procedures with the following exceptions:

1. Silver recoveries were low and could be due to AgCl precipitation during sample preparation. Additionally, adsorption of silver to digestate container walls is possible.
2. The Cr spike in the "Total" digestate was high. This was probably because of the relatively high quantities of Cr in the sample compared to the spike level.

TABLE 4-1 --Physical Characteristics and Percent Moisture of Study Samples

Sample	Physical Characteristics	Percent Solids
Sn Smelter Slag	Dark grey, dense, equal mixture of fine powder and gravel	99.8
Pb/Zn Smelter Slag	Dark grey to black, porous,gravel	99.9
Cu Smelter Slag	Reddish brown, porous, gravel	99.9
Pb Smelter Slag	Black, porous, sand to gravel	98.5
EPA Preaward #1	Brownish, silty clay	47.4

Table 4-2 Element Concentrations in the 3050, "Total", and EP

(C o n c e n t r a t i o n - mg/L)

	Arsenic			Barium			Cadmium			Chromium			Lead			Silver		
Sample ID	3050	"Total"	EP	3050	"Total"	EP	3050	"Total"	EP	3050	"Total"	EP	3050	"Total"	EP	3050	"Total"	EP
Sn Smelter Slag	<50*	<200*	<0.05	810	912	0.435	<4*	1.0	0.006	109	1170	0.005	144	144	0.071	<10*	<40*	<0.01
Pb/Zn Smelter Slag	288	144	<0.05	3060	3440	2.84	26	20.4	0.035	<8*	112	<0.004	17600	18500	35.7	<20*	<40*	<0.01
Cu Smelter Slag	<5	<200*	<0.05	5.1	376	0.075	<0.4	3.2	0.004	4.2	276	<0.004	<5	26.4	<0.05	<1	<40*	<0.01
Pb Smelter Slag	<50*	<200*	<0.05	190	260	0.041	70	72	2.34	36	412	<0.004	31400	29000	352	<20*	<40*	<0.01
EPA Preaward #1	13700	12800	1.56	13200	13240	0.62	26400	25200	556	11000	11000	0.48	113000	92000	15.5	83	17.6	0.061
EP Toxicity MCL			5.0			100			1.0			5.0			5.0			5.0

*Sample was diluted, which resulted in increased detection limit.

3. Arsenic recovery was low in the "Total" digestate. A loss of As in the preparation procedure, due to its volatility, is suspected.

Duplicate results were generally quite good (less than 20 percent RPD). There were a few exceptions, the most notable of which was the 89 percent RPD for lead in Sn slag after the 3050 digestion. This variation may be due to a non-homogeneous sample, or contamination during sample preparation.

QC data are provided in detail in Appendix 2.

All samples were bench spiked to determine the accuracy of the ICP method on these sample types (see Appendix 1). As can be seen in Appendix 1, 57 of 64 spike recoveries for the EP, 64 of 72 for the "Total," and 67 of 72 for the 3050 were in the 80 to 120 percent recovery range. This indicates that the ICP method is providing an acceptable data base without any inherent biases for these sample types.

To supplement the quality control procedures used in the method, an EPA- EP-QC Sludge (Preaward Sample # 1) was obtained and analyzed by all the preparation schemes. Table 4-3 compares the EPA-EP acceptable ranges to the observed EP-values for this sample. As can be seen in the table, several elements fell outside the "Acceptance Range". EPA has not "certified" these elements, however, and is investigating the differences between the "true" and observed levels (communication with Ms. Florence Richardson, EPA-OSW).

Table 4-3 EPA EP-Preaward #1 -
Reference Value, Acceptable Ranges, and Observed Value

Element	Observed value	Reference value	Acceptance range
Ag*	0.061	0.12	0.06 - 0.24
As*	1.56	12.8	6.4 - 25.6
Ba*	0.62	2.4	1.2 - 4.8
Ca	76.6	83.2	41.6 - 166.4
Cd	556.0	717.5	358.8 - 1435.0
Cr*	0.48	0.16	0.08 - 0.32
Hg*	-----	128.0	64.0 - 256.0
K	732.0	827.0	413.5 - 1654.0
Mg	86.0	99.7	49.8 - 199.4
Na	532.0	560.0	280.0 - 1120.0
Ni	69.0	78.4	39.2 - 156.8
Pb*	15.5	69.2	34.6 - 138.4
Se	-----	2.4	1.2 - 4.8
Zn	320.0	362.0	181.0 - 724.0

* Values for these elements are not "certified." EPA, according to Ms. Florence Richardson, is investigating difficulties between the "true" and observed levels of these elements.

4.1.2 Task 1 Discussion

Method 3050 does not totally digest a solid sample, but is really an acid leaching procedure. Visible solids remained after the digestion, as well as what appeared to be a gelatinous layer in the four mining waste samples. Repeating the digestion of one sample using less sample weight but the same quantities of acids was undertaken to see if a more thorough digestion of the gelatinous material was possible. Results indicated that as the sample weight was decreased in the digestion, slightly higher metal concentrations were observed.

For comparison with the 3050 digestion, a "Total" metals digestion was carried out with HF:HNO3:HClO4 acids. No visible solids remained after this digestion, thus indicating that this was a complete digestion.

In general, for the six elements of interest, the data were roughly equivalent between the two acid digestions. Chromium was an exception, where considerably higher concentrations were observed in the "Total" digestate. This seems to indicate that five of the six elements were bound in more easily released phases in these samples.

The objective of Task 1 was to compare EP-leachate metal values for mining and smelting wastes relative to total values. The comparison showed that, for all six metals, there was a sufficient quantity of metal in each sample to account for the EP results. This can be seen for Pb and Cd in Table 4-4, which compares data in the EP-leachate with the amount that could be potentially extracted from the sample based on the 3050 digestate levels. The data base, however, is limited and therefore may not be indicative of all mining and smelting waste samples.

Table 4-4 Comparison of Metals Released by 3050 Digestion With the EP-Toxicity Test

	Cadmium			Lead		
	3050 mg/kg	Potential* EP mg/l	EP mg/l	3050 mg/l	Potential* EP mg/l	EP mg/l
Sn smelter slag	<5.	<0.25	<0.05	144	7.2	0.071
Pb/Zn smelter slag	26	1.3	<0.05	17,600	880	35.7
Cu smelter slag	<5.	<0.25	<0.05	<5	<0.25	<0.05
Pb smelter slag	70	3.4	2.34	31,400	1,550	352
EPA preaward #1	26,400	626	556	113,000	2,680	15.5

* The Potential EP is the concentration expected in an EP, if all the metal were released: 3050 value (mg/kg) x percent solids (see Table 4-1) x 0.1 kg total / EP-extract volume (2 L for all samples except EPA sample, which was 1.809 L).

4.2 Task 2 - Comparisons of Four Extraction Techniques

4.2.1 Task 2 Results and Quality Control Data

The data for samples in the SRL, EP-w/o, and ASTM extractions (EP results were discussed in the previous section and are also included in Appendix I) including bench spike, duplicate, and blank results can be found in Appendix 1. Most bench spike recoveries were within the 80 to 120 percent range, indicating accurate analyte quantitation. Exceptions outside of this range are as follows:

Extraction	Sample ID	Metal(s)
EP-w/o	Pb/Zn Slag	As, Cr, Pb
SRL	Cu Slag	Pb
SRL	EPA Preaward 1	Ba
ASTM	Pb/Zn Slag	As
ASTM	EPA Preaward 1	Ba, Cd

Other QC data on calibration blank values, calibration check standard results, and duplicate RSD are reported in Appendix 2. Of note here is the relatively poor precision found for duplicates in the SRL. More detailed study of this new procedure would be necessary to determine whether the poor precision is inherent in the method or a result of these particular samples.

Table 4-5 compares data on the five samples by element and extraction procedure. EP-maximum contaminant levels (MCL) are also provided in this table. The EPA Preaward #1 and Pb Smelter Slag exceeded MCLs for both Cd and Pb, and sample Pb/Zn Smelter Slag exceeded the MCL for

Table 4-5 Element Concentrations in the Four Extractions

(C o n c e n t r a t i o n - mg/L)

Sample ID	Arsenic EP	Arsenic EP w/o	Arsenic ASTM	Arsenic SRL	Barium EP	Barium EP w/o	Barium ASTM	Barium SRL	Cadmium EP	Cadmium EP w/o	Cadmium ASTM	Cadmium SRL
Sn Smelter Slag	<0.05	<0.05	<0.05	<0.05	0.435	0.095	0.063	1.18	0.006	<0.004	<0.004	<0.004
Pb/Zn Smelter Slag	<0.05	<0.05	<0.05	<0.5 *	2.84	0.363	0.217	7.56	0.035	<0.004	0.065	0.02
Cu Smelter Slag	<0.05	<0.05	<0.05	<0.05	0.075	0.047	0.042	0.769	0.004	<0.004	<0.004	<0.004
Pb Smelter Slag	<0.05	<0.05	<0.05	<1 *	0.041	0.054	0.039	1.04	2.34	1.08	1.98	1.4
EPA Preaward #1	1.56	0.073	0.55	140	0.62	0.281	0.46	0.58	556	8.4	20	594
EP Toxicity MCL	5.0				100				1.0			

Sample ID	Chromium EP	Chromium EP w/o	Chromium ASTM	Chromium SRL	Lead EP	Lead EP w/o	Lead ASTM	Lead SRL	Silver EP	Silver EP w/o	Silver ASTM	Silver SRL
Sn Smelter Slag	0.005	0.007	0.008	0.025	0.071	<0.05	<0.05	0.632	<0.01	<0.01	<0.01	<0.01
Pb/Zn Smelter Slag	<0.004	<0.004	<0.004	0.006	35.7	<0.05	8.94	55.2	<0.01	<0.01	<0.01	<0.2 *
Cu Smelter Slag	<0.004	<0.004	<0.004	0.005	<0.05	<0.05	<0.05	0.112	<0.01	<0.01	<0.01	<0.01
Pb Smelter Slag	<0.004	<0.004	0.005	0.025	352	0.054	0.039	222	<0.01	<0.01	<0.01	<0.2 *
EPA Preaward #1	0.48	0.011	0.24	61.4	15.5	0.236	3.3	868	0.061	0.028	0.146	0.063
EP Toxicity MCL	5.0				5.0				5.0			

* Sample was diluted, which resulted in increased detection limits.

Pb only. No other MCLs were exceeded by these samples for the six elements of interest.

4.2.2 Task 2 Discussion

For most elements, the SRL data was greater than or equal to EP data. The data for these two extractions were generally much greater than the ASTM and EP-w/o data. The ASTM had levels slightly greater than those found in the EP-w/o. There were exceptions (As, Ag, Cr) where insufficient data (i.e., metal levels less than detection limits) inhibited an accurate portrayal of these trends. This relationship was more obvious in comparison of some of the major elements (Fe, Ca, Mg, Na, Zn) that were quantitated concurrently by the ICP method. The data for these elements are provided in Table 4-6.

The higher level of elements in the ASTM extract over the EP-w/o would be expected because of the larger solid to water ratio (i.e., ASTM was 1:4 solid:water; EP-w/o was 1:20). The larger values in the other two extractions are not as easily understood, but may be related to the pH of the extraction fluid. As the pH decreases sample dissolution (e.g., metal sulfides and carbonates, colloidal material) or possibly organic matter destruction could occur, with resultant leachate metal concentration increases. Additionally, cation replacement on surface exchange sites by H+ would increase as the pH decreases. Table 4-7 reports initial and final pH values in the four extracts. As can be seen by comparing the pH data with the metals data in the extracts, the lowest pH values were in the SRL (pH of extraction fluid 3.98) where the

Table 4-6 Other Major Element Concentrations in the Four Extractions

(Concentration - mg/L)

Sample ID	Calcium				Iron				Magnesium			
	EP	EP w/o	ASTM	SRL	EP	EP w/o	ASTM	SRL	EP	EP w/o	ASTM	SRL
Sn Smelter Slag	59.8	6.21	22.1	82.1	28.2	0.13	0.225	59.9	18.3	2.46	7.15	13.7
Pb/Zn Smelter Slag	140	4.85	3.57	268	13	0.024	1.05	171	6.64	0.266	0.224	16.9
Cu Smelter Slag	1.43	1.08	5.14	2.16	2.39	0.181	0.189	27.9	0.374	0.256	1.77	0.818
Pb Smelter Slag	24.8	13	57.8	181	1.59	0.022	0.239	386	5.48	1.44	4.29	58
EPA Preaward #1	67.9	33.7	106	54.8	0.143	0.042	0.398	20.5	86.2	50.5	236	67.8

Sample ID	Sodium				Zinc			
	EP	EP w/o	ASTM	SRL	EP	EP w/o	ASTM	SRL
Sn Smelter Slag	14	12.6	65.4	19.6	5.62	0.041	0.049	6.65
Pb/Zn Smelter Slag	4.1	0.947	2.04	12.1	102	0.026	6.36	324
Cu Smelter Slag	1.2	1.44	8.34	5.17	0.112	0.027	0.034	0.532
Pb Smelter Slag	2.4	0.946	3.67	22.1	159	11.3	8.5	264
EPA Preaward #1	532	326	1620	404	312	7.36	12.6	458

Table 4-7 Initial and Final pH Measurements in Extracts

	pH Measurements							
	EP		EP-w/o		ASTM		SRL	
Sample no.	Initial	Final	Initial	Final	Initial	Final	Initial	Final
EPA pre-award #1	7.51*	4.94	7.51	7.15	7.51	7.14	-**	7.05
2023	8.72*	4.90	8.78	9.76	8.80	8.49	-**	5.67
2024	6.48*	4.81	6.51	9.93	6.40	6.75	-**	5.18
2025	6.55*	5.18	6.56	8.73	6.56	8.78	-**	4.92
2028	6.52*	5.10	6.53	6.31	6.53	6.24	-**	5.63

* pH adjusted to 5.0 with 0.5 N acetic acid.

** Initial pH was not measured; however, pH of the extraction fluid was 3.98.

highest metal concentrations were found. The EP pH values were higher, as were respective metal concentrations. Conversely, where the highest pHs were found, the lowest metal values were observed (EP-w/o and the ASTM). This indicates that the pH of the extraction fluid is a controlling factor in metal leaching from samples, and not the suspected affinity of acetic acid for metals such as Pb and Cd.

It is difficult to ascertain whether a laboratory technique can realistically predict the release of these metals in situ. It would appear that the acidity of the rainfall at a dumpsite could control the release of metals from wastes. Since rainfall acidity varies geographically, it may be impossible to design an absolute test for use in every situation. The EP-Toxicity Test will continue to serve as a useful screening procedure for identifying wastes which are potentially hazardous when disposed of in certain environments. However, the Agency should develop additional screening tests and procedures, including one that simulates the effect of acid precipitation on mine and smelter wastes.

APPENDIX 1

ICP ANALYTICAL DATA

KEY PHRASES FOR APPENDIX 1 TABLES

KEY:

Digestion DF = Dilution factor in acid digestions.

Unadj. Conc. = Unadjusted Concentration readout from ICP.
Dilution Factor = Extract dilution factor prior to ICP quantitation.
Reported Value = Final reported value with dilution factors applied.

Spike Added = Bench spike added prior to ICP analysis.
Obs. Spk. Value = Observed ICP value of bench spiked sample.
% Recovery = Bench spike recovery.

Preparation Procedure: Method 3050

Date: 29-May-86

CONCENTRATION

Sample No.		As	Ba	Cd	Cr	Pb	Ag	units
Blank	*Digestion DF = 1							
	Unadj. Conc.	<0.05	<0.003	<0.004	<0.004	<0.05	<0.01	mg/L
	Dilution Factor	1	1	1	1	1	1	
	Reported Value	<0.05	<0.003	<0.004	<0.004	<0.05	<0.01	mg/L
	Spike Added	0.2	0.1	0.1	0.1	0.2	0.2	mg/L
	Obs. Spk. Value	0.178	0.1	0.092	0.099	0.21	0.181	mg/L
	% Recovery	89%	100%	92%	99%	105%	91%	
Sn Smelter Slag	*Digestion DF =100							
	Unadj. Conc.	<0.05	0.81	<0.004	1.09	1.44	<0.01	mg/L
	Dilution Factor	10	10	10	1	1	10	
	Reported Value	<50	810	<4	109	144	<10	mg/kg
	Spike Added	200	1500	200	200	300	200	mg/kg
	Obs. Spk. Value	184	2120	204	283	396	169	mg/kg
	% Recovery	92%	87%	102%	87%	84%	85%	
Sn Smelter Slag duplicate	*Digestion DF =100							
	Unadj. Conc.	<0.05	0.718	<0.004	0.119	0.375	<0.01	mg/L
	Dilution Factor	10	10	10	10	10	10	
	Reported Value	<50	718	<4	119	375	<10	mg/kg
	Spike Added	100	15000	100	2000	6000	200	mg/kg
	Obs. Spk. Value	137	16600	100	2240	6470	172	mg/kg
	% Recovery	137%	106%	100%	106%	102%	86%	
Pb/Zn Smelter Slag	*Digestion DF = 100							
	Unadj. Conc.	0.144	1.53	0.013	<0.004	8.8	<0.01	mg/L
	Dilution Factor	20	20	20	20	20	20	
	Reported Value	288	3060	26	<8	17600	<20	mg/kg
	Spike Added	10000	100000	1000	200	700000	400	mg/kg
	Obs. Spk. Value	11400	118000	1050	198	808000	366	mg/kg
	% Recovery	111%	115%	102%	99%	113%	92%	

* Digestion Dilution Factor = 1.0gm/100mL

Preparation Procedure: Method 3050

Date: 29-May-86

CONCENTRATION

Sample No.	As	Ba	Cd	Cr	Pb	Ag	units
Pb/Zn Smelter Slag duplicate							
*Digestion DF =100							
Unadj. Conc.	0.158	1.49	0.014	<0.004	7.66	<0.01	mg/L
Dilution Factor	20	20	20	1	20	1	
Reported Value	316	2980	28	<0.4	15300	<1	mg/kg
Spike Added	10000	100000	1000	200	600000	400	mg/kg
Obs. Spk. Value	11500	123000	980	208	752000	350	mg/kg
% Recovery	112%	120%	95%	104%	123%	88%	
Pb/Zn Smelter Slag spike							
*Digestion DF =100							
Unadj. Conc.	0.207	2.52	0.021	0.005	12.3	<0.01	mg/L
Dilution Factor	20	20	20	20	20	20	
Reported Value	414	5040	42	10	24600	<20	mg/kg
Spike Added	14000	200000	1500	300	1000000	400	mg/kg
Obs. Spk. Value	16000	204000	1540	360	1190000	354	mg/kg
% Recovery	111%	99%	100%	117%	117%	89%	
Cu Smelter Slag							
*Digestion DF =100							
Unadj. Conc.	<0.05	0.051	<0.004	0.042	<0.05	<0.01	mg/L
Dilution Factor	1	1	20	1	1	1	
Reported Value	<5	5.1	<0.4	4.2	<5	<1	mg/kg
Spike Added	400	400	200	400	400	400	mg/kg
Obs. Spk. Value	412	412	216	416	396	372	mg/kg
% Recovery	103%	102%	108%	103%	99%	93%	
Cu Smelter Slag duplicate							
*Digestion DF =100							
Unadj. Conc.	<0.05	0.065	<0.004	0.073	<0.05	<0.01	mg/L
Dilution Factor	20	1	20	1	20	20	
Reported Value	<50	6.5	<8	7.3	<100	<20	mg/kg
Spike Added	400	400	200	400	400	400	mg/kg
Obs. Spk. Value	408	408	226	414	400	348	mg/kg
% Recovery	102%	100%	113%	102%	100%	87%	

* Digestion Dilution Factor = 1.0gm/100mL

Preparation Procedure: Method 3050

Date: 29-May-86

CONCENTRATION

Sample No.	As	Ba	Cd	Cr	Pb	Ag	units
Pb Smelter Slag							
*Digestion DF =100							
Unadj. Conc.	<0.05	0.095	0.035	0.018	15.7	<0.01	mg/L
Dilution Factor	20	20	20	20	20	20	
Reported Value	<50	190	70	36	31400	<20	mg/kg
Spike Added	400	7000	2400	1200	1200000	400	mg/kg
Obs. Spk. Value	586	7500	2700	1280	1620000	382	mg/kg
% Recovery	147%	104%	110%	104%	132%	96%	
Pb Smelter Slag duplicate							
*Digestion DF =100							
Unadj. Conc.	<0.05	0.098	0.041	0.02	16.9	<0.01	mg/L
Dilution Factor	20	20	20	20	20	20	
Reported Value	<50	196	82	40	33800	<20	mg/kg
Spike Added	400	7000	2400	1200	1200000	400	mg/kg
Obs. Spk. Value	572	7320	2740	1280	1460000	364	mg/kg
% Recovery	143%	102%	111%	103%	119%	91%	
EPA Preaward #1							
*Digestion DF =100							
Unadj. Conc.	13.7	13.2	26.4	11	56.6	0.083	mg/L
Dilution Factor	10	10	10	10	20	10	
Reported Value	13700	13200	26400	11000	113000	83	mg/kg
Spike Added	25000	25000	50000	25000	4400000	1600	mg/kg
Obs. Spk. Value	36400	36900	72400	34600	4860000	1370	mg/kg
% Recovery	91%	95%	92%	94%	108%	80%	
EPA Preaward #1 duplicate							
*Digestion DF =100							
Unadj. Conc.	7.21	7	13.8	5.94	58.6	0.042	mg/L
Dilution Factor	20	20	20	20	20	20	
Reported Value	14400	14000	27600	11900	117000	84	mg/kg
Spike Added	50000	50000	100000	50000	4120000	3400	mg/kg
Obs. Spk. Value	66000	62400	123000	59800	4760000	2660	mg/kg
% Recovery	103%	97%	95%	96%	113%	76%	

* Digestion Dilution Factor = 1.0gm/100mL

Preparation Procedure: Total Digestion

Date: 28-May-86

Sample No.		As	Ba	Cd	Cr	Pb	Ag	units
		CONCENTRATION						
Blank								
	*Digestion DF = 1							
	Unadj. Conc.	<0.05	0.122	<0.004	0.005	0.057	<0.01	mg/L
	Dilution Factor	1	1	1	1	1	1	
	Reported Value	<0.05	0.122	<0.004	0.005	0.057	<0.01	mg/L
	Spike Added	0.1	0.25	0.1	0.1	0.2	0.2	mg/L
	Obs. Spk. Value	0.091	0.37	0.102	0.105	0.238	0.178	mg/L
	% Recovery	91%	99%	102%	100%	91%	89%	
Sn Smelter Slag								
	*Digestion DF = 200							
	Unadj. Conc.	<0.05	0.228	0.005	0.292	0.722	<0.01	mg/L
	Dilution Factor	20	20	1	20	1	20	
	Reported Value	<200	912	1	1170	144	<40	mg/kg
	Spike Added	800	36000	400	40000	6000	800	mg/kg
	Obs. Spk. Value	856	38000	436	42800	6360	644	mg/kg
	% Recovery	107%	103%	109%	104%	104%	81%	
Sn Smelter Slag duplicate								
	*Digestion DF =200							
	Unadj. Conc.	<0.05	0.23	0.009	0.285	0.748	<0.01	mg/L
	Dilution Factor	20	20	1	20	1	20	
	Reported Value	<200	920	1.80	1140	150	<40	mg/kg
	Spike Added	800	36000	400	40000	6000	800	mg/kg
	Obs. Spk. Value	812	38800	428	44000	6440	680	mg/kg
	% Recovery	102%	105%	107%	107%	105%	85%	
Pb/Zn Smelter Slag								
	*Digestion DF =200							
	Unadj. Conc.	<0.05	0.86	0.102	0.028	4.62	<0.01	mg/L
	Dilution Factor	20	20	1	20	20	20	
	Reported Value	<200	3440	20.4	112	18500	<40	mg/kg
	Spike Added	6000	128000	800	4000	680000	800	mg/kg
	Obs. Spk. Value	6920	144000	880	4720	756000	684	mg/kg
	% Recovery	115%	110%	107%	115%	108%	86%	

* Digestion Dilution Factor = 0.5gm/100ml

Preparation Procedure: Total Digestion

Date: 28-May-86

CONCENTRATION

Sample No.	As	Ba	Cd	Cr	Pb	Ag	units
Pb/Zn Smelter Slag duplicate *Digestion DF =200							
Unadj. Conc.	0.762	0.897	0.006	0.03	5.12	<0.01	mg/L
Dilution Factor	1	20	20	20	20	20	
Reported Value	152	3590	24	120	20500	<40	mg/kg
Spike Added	6000	128000	800	4000	680000	800	mg/kg
Obs. Spk. Value	7200	144000	860	4760	760000	672	mg/kg
% Recovery	117%	110%	105%	116%	109%	84%	
Pb/Zn Smelter Slag spike *Digestion DF =200							
Unadj. Conc.	0.073	1.83	0.011	0.04	8.34	0.025	mg/L
Dilution Factor	20	20	20	20	20	1	
Reported Value	292	7320	44	160	33400	5.0	mg/kg
Spike Added	12000	280000	1600	6000	1200000	800	mg/kg
Obs. Spk. Value	14200	316000	1770	6920	1630000	712	mg/kg
% Recovery	116%	110%	108%	113%	133%	88%	
Cu Smelter Slag *Digestion DF =200							
Unadj. Conc.	<0.05	0.094	0.016	0.069	0.132	<0.01	mg/L
Dilution Factor	20	20	1	20	1	20	
Reported Value	<200	376	3.2	276	26.4	<40	mg/kg
Spike Added	400	16000	400	12000	1200	800	mg/kg
Obs. Spk. Value	456	21900	396	15400	1150	712	mg/kg
% Recovery	114%	135%	98%	126%	94%	89%	
Cu Smelter Slag duplicate *Digestion DF =200							
Unadj. Conc.	<0.05	0.096	0.018	0.072	<0.05	<0.01	mg/L
Dilution Factor	20	20	1	20	1	20	
Reported Value	<200	384	3.6	288	<10	<40	mg/kg
Spike Added	400	16000	400	12000	1200	800	mg/kg
Obs. Spk. Value	396	22300	424	15300	1130	676	mg/kg
% Recovery	99%	137%	105%	125%	94%	85%	

* Digestion Dilution Factor = 0.5gm/100ml

Preparation Procedure: Total Digestion

Date: 28-May-86

CONCENTRATION

Sample No.	As	Ba	Cd	Cr	Pb	Ag	units
Pb Smelter Slag *Digestion DF =200							
Unadj. Conc.	<0.05	0.065	0.018	0.103	7.26	<0.01	mg/L
Dilution Factor	20	20	20	20	20	20	
Reported Value	<200	260	72	412	29000	<40	mg/kg
Spike Added	800	12000	2400	14000	1000000	800	mg/kg
Obs. Spk. Value	1030	16900	2490	17800	1470000	668	mg/kg
% Recovery	129%	139%	101%	124%	144%	84%	
Pb Smelter Slag duplicate *Digestion DF =200							
Unadj. Conc.	<0.05	0.07	0.02	0.102	7.13	<0.01	mg/L
Dilution Factor	20	20	20	20	20	20	
Reported Value	<200	280	80	408	28500	<40	mg/kg
Spike Added	800	12000	2400	14000	1000000	800	mg/kg
Obs. Spk. Value	952	12300	2430	14700	1080000	692	mg/kg
% Recovery	119%	100%	98%	102%	105%	87%	
EPA Preaward #1 *Digestion DF =200							
Unadj. Conc.	3.2	3.31	6.29	2.74	23	0.088	mg/L
Dilution Factor	20	20	20	20	20	1	
Reported Value	12800	13200	25200	11000	92000	17.6	mg/kg
Spike Added	28000	28000	52000	24000	200000	800	mg/kg
Obs. Spk. Value	43200	42400	83200	36000	305000	732	mg/kg
% Recovery	109%	104%	112%	104%	107%	89%	
EPA Preaward #1 duplicate *Digestion DF =200							
Unadj. Conc.	3.39	2	6.24	2.62	27.3	0.04	mg/L
Dilution Factor	20	20	20	20	20	1	
Reported Value	13600	8000	25000	10500	109000	8.0	mg/kg
Spike Added	28000	16000	52000	24000	220000	800	mg/kg
Obs. Spk. Value	44400	24600	82400	35400	342000	660	mg/kg
% Recovery	110%	104%	110%	104%	106%	82%	

* Digestion Dilution Factor = 0.5gm/100ml

Preparation Procedure: EP TOXICITY TEST

Date: 29-May-86 Page: 1 of 3

Sample No.	CONCENTRATION (units = mg/L) As	Ba	Cd	Cr	Pb	Ag
Blank						
Unadj. Conc.	<0.05	<0.003	<0.004	<0.004	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	<0.003	<0.004	<0.004	<0.05	<0.01
Spike Added	0.1	0.1	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.097	0.098	0.094	0.096	0.164	0.168
% Recovery	97%	98%	94%	96%	82%	84%
Sn Smelter Slag						
Unadj. Conc.	<0.05	0.435	0.006	0.005	0.071	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.435	0.006	0.005	0.071	<0.01
Spike Added	0.1	1	0.1	0.1	0.3	0.2
Obs. Spk. Value	0.089	1.45	0.101	0.098	0.335	0.172
% Recovery	89%	102%	95%	93%	88%	86%
Sn Smelter Slag duplicate						
Unadj. Conc.	<0.05	0.431	<0.004	<0.004	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.431	<0.004	<0.004	<0.05	<0.01
Spike Added	0.2	1	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.183	1.45	0.094	0.1	0.197	0.169
% Recovery	92%	102%	94%	100%	99%	85%
Pb/Zn Smelter Slag						
Unadj. Conc.	<0.05	2.84	0.035	<0.004	35.7	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	2.84	0.035	<0.004	35.7	<0.01
Spike Added	0.2	6	0.1	0.1	70	0.2
Obs. Spk. Value	0.218	8.59	0.128	0.095	103	0.167
% Recovery	109%	96%	93%	95%	96%	84%

Preparation Procedure: EP TOXICITY TEST

Date: 29-May-86

Sample No.	CONCENTRATION (units = mg/L) As	Ba	Cd	Cr	Pb	Ag
Pb/Zn Smelter Slag duplicate						
Unadj. Conc.	<0.05	2.67	0.037	<0.004	36.9	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	2.67	0.037	<0.004	36.9	<0.01
Spike Added	0.2	6	0.1	0.1	70	0.2
Obs. Spk. Value	0.228	8.64	0.132	0.092	105	0.167
% Recovery	114%	100%	95%	92%	97%	84%
Cu Smelter Slag						
Unadj. Conc.	<0.05	0.075	<0.004	<0.004	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.075	<0.004	<0.004	<0.05	<0.01
Spike Added	0.2	0.2	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.165	0.259	0.094	0.098	0.177	0.17
% Recovery	83%	92%	94%	98%	89%	85%
Cu Smelter Slag duplicate						
Unadj. Conc.	<0.05	0.047	0.009	<0.004	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.047	0.009	<0.004	<0.05	<0.01
Spike Added	0.2	0.2	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.179	0.232	0.097	0.098	0.175	0.164
% Recovery	90%	93%	88%	98%	88%	82%
Pb Smelter Slag						
Unadj. Conc.	<0.05	0.041	0.117	<0.004	17.6	<0.01
Dilution Factor	1	1	20	1	20	1
Reported Value	<0.05	0.041	2.34	<0.004	352	<0.01
Spike Added	4	2	80	2	12000	4
Obs. Spk. Value	5.06	2.04	93.4	1.98	13760	3.42
% Recovery	127%	98%	114%	99%	112%	86%

Preparation Procedure: EP TOXICITY TEST

Date: 29-May-86

Sample No.	CONCENTRATION (units = mg/L) As	Ba	Cd	Cr	Pb	Ag
Pb Smelter Slag duplicate						
Unadj. Conc.	<0.05	0.057	0.087	<0.004	21.2	<0.01
Dilution Factor	1	1	20	1	20	1
Reported Value	<0.05	0.057	1.74	<0.004	424	<0.01
Spike Added	4	2	60	2	16000	4
Obs. Spk. Value	5.26	2.04	70.2	2.04	18800	3.14
% Recovery	132%	99%	114%	102%	115%	79%
EPA Preaward #1						
Unadj. Conc.	0.078	0.031	27.8	0.024	0.773	0.061
Dilution Factor	20	20	20	20	20	1
Reported Value	1.56	0.62	556	0.48	15.5	0.061
Spike Added	60	22	22000	20	540	4
Obs. Spk. Value	77.2	28.8	23500	22.8	776	3.48
% Recovery	126%	128%	104%	112%	141%	85%
EPA Preaward #1 duplicate						
Unadj. Conc.	0.081	0.032	27.2	0.026	0.807	0.054
Dilution Factor	20	20	20	20	20	1
Reported Value	1.62	0.64	544	0.52	16.1	0.054
Spike Added	60	22	22000	20	580	4
Obs. Spk. Value	63.8	29	23800	23.6	620	3.56
% Recovery	104%	129%	106%	115%	104%	88%

Preparation Procedure: EP TOXICITY TEST WITHOUT pH ADJUSTMENT

Date: 29-May-86

Sample No.	CONCENTRATION (units = mg/L) As	Ba	Cd	Cr	Pb	Ag
Blank						
Unadj. Conc.	<0.05	<0.003	<0.004	<0.004	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	<0.003	<0.004	<0.004	<0.05	<0.01
Spike Added	0.1	0.1	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.104	0.1	0.097	0.102	0.223	0.181
% Recovery	104%	100%	97%	102%	112%	91%
Sn Smelter Slag						
Unadj. Conc.	<0.05	0.095	<0.004	0.007	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.095	<0.004	0.007	<0.05	<0.01
Spike Added	0.1	0.2	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.098	0.295	0.093	0.103	0.179	0.174
% Recovery	98%	100%	93%	96%	90%	87%
Sn Smelter Slag duplicate						
Unadj. Conc.	<0.05	0.112	<0.004	0.007	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.112	<0.004	0.007	<0.05	<0.01
Spike Added	0.1	0.2	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.117	0.3	0.095	0.106	0.185	0.176
% Recovery	117%	94%	95%	99%	93%	88%
Pb/Zn Smelter Slag						
Unadj. Conc.	<0.05	0.363	<0.004	<0.004	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.363	<0.004	<0.004	<0.05	<0.01
Spike Added	0.1	0.75	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.223	1.13	0.106	0.144	0.574	0.213
% Recovery	223%	102%	106%	144%	287%	107%

Preparation Procedure: EP TOXICITY TEST WITHOUT pH ADJUSTMENT

Date: 29-May-86

Sample No.	CONCENTRATION (units = mg/L) As	Ba	Cd	Cr	Pb	Ag
Cu Smelter Slag						
Unadj. Conc.	<0.05	0.047	<0.004	<0.004	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.047	<0.004	<0.004	<0.05	<0.01
Spike Added	0.1	0.1	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.105	0.147	0.1	0.104	0.182	0.18
% Recovery	105%	100%	100%	104%	91%	90%
Pb Smelter Slag						
Unadj. Conc.	<0.05	0.054	1.08	<0.004	4.65	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.054	1.08	<0.004	4.65	<0.01
Spike Added	0.1	0.1	2	0.1	10	0.2
Obs. Spk. Value	0.1	0.152	3.43	0.111	14.1	0.181
% Recovery	100%	98%	118%	111%	95%	91%
EPA Preaward #1						
Unadj. Conc.	0.073	0.281	8.4	0.011	0.236	0.028
Dilution Factor	1	1	1	1	1	1
Reported Value	0.073	0.281	8.4	0.011	0.236	0.028
Spike Added	0.2	0.6	17	0.1	0.5	0.2
Obs. Spk. Value	0.253	0.826	27.8	0.102	0.663	0.206
% Recovery	90%	91%	114%	91%	85%	89%

Preparation Procedure: ASTM EXTRACTION

Date: 29-May-86

Sample No.	CONCENTRATION (units = mg/L) As	Ba	Cd	Cr	Pb	Ag
Blank						
Unadj. Conc.	<0.05	<0.003	0.005	<0.004	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	<0.003	0.005	<0.004	<0.05	<0.01
Spike Added	0.1	0.1	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.097	0.1	0.099	0.104	0.199	0.178
% Recovery	97%	100%	94%	104%	100%	89%
Sn Smelter Slag						
Unadj. Conc.	0.05	0.063	<0.004	0.008	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	0.05	0.063	<0.004	0.008	<0.05	<0.01
Spike Added	0.1	0.15	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.146	0.211	0.098	0.105	0.181	0.176
% Recovery	96%	99%	98%	97%	91%	88%
Pb/Zn Smelter Slag						
Unadj. Conc.	<0.05	0.217	0.065	<0.004	8.94	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.217	0.065	<0.004	8.94	<0.01
Spike Added	0.1	0.4	0.1	0.1	18	0.2
Obs. Spk. Value	0.133	0.607	0.165	0.101	30.4	0.187
% Recovery	133%	98%	100%	101%	119%	94%
Cr Smelter Slag						
Unadj. Conc.	<0.05	0.042	<0.004	<0.004	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.042	<0.004	<0.004	<0.05	<0.01
Spike Added	0.1	0.1	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.081	0.14	0.095	0.101	0.183	0.178
% Recovery	81%	98%	95%	101%	92%	89%

Preparation Procedure: ASTM EXTRACTION

Date: 29-May-86

Sample No.	CONCENTRATION (units = mg/L) As	Ba	Cd	Cr	Pb	Ag
Cu Smelter Slag duplicate						
Unadj. Conc.	<0.05	0.032	<0.004	0.006	<0.05	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.032	<0.004	0.006	<0.05	<0.01
Spike Added	0.2	0.1	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.17	0.132	0.1	0.117	0.19	0.181
% Recovery	85%	100%	100%	111%	95%	91%
Pb Smelter Slag						
Unadj. Conc.	<0.05	0.039	1.98	0.005	3.64	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.039	1.98	0.005	3.64	<0.01
Spike Added	0.2	0.1	4	0.1	8	0.2
Obs. Spk. Value	0.182	0.138	8.36	0.104	13.7	0.184
% Recovery	91%	98%	114%	99%	112%	92%
EPA Preaward #1						
Unadj. Conc.	0.55	0.023	1.0	0.012	0.165	0.146
Dilution Factor	1	20	20	20	20	1
Reported Value	0.55	0.46	20	0.24	3.3	0.146
Spike Added	22	20	800	10	120	6
Obs. Spk. Value	26.3	29	1200	10.1	137	5.36
% Recovery	117%	143%	148%	99%	111%	87%

Preparation Procedure: SYNTHETIC RAINWATER LEACH (SRL)

Date: 29-May-86

Sample No.	CONCENTRATION (units = mg/L) As	Ba	Cd	Cr	Pb	Ag
Blank						
Unadj. Conc.	<0.05	<0.003	<0.004	<0.004	<0.05	<0.001
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	<0.003	<0.004	<0.004	<0.05	<0.001
Spike Added	0.2	0.1	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.192	0.099	0.095	0.096	0.169	0.177
% Recovery	96%	99%	95%	96%	85%	89%
Sn Smelter Slag						
Unadj. Conc.	<0.05	1.18	<0.004	0.025	0.632	<0.001
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	1.18	<0.004	0.025	0.632	<0.001
Spike Added	0.2	3	0.1	0.1	1.3	0.2
Obs. Spk. Value	0.21	4.03	0.098	0.123	1.84	0.17
% Recovery	105%	95%	98%	98%	93%	85%
Pb/Zn Smelter Slag						
Unadj. Conc.	<0.05	0.378	0.02	0.006	2.76	<0.001
Dilution Factor	20	20	1	1	20	20
Reported Value	<0.5	7.56	0.02	0.006	55.2	<0.2
Spike Added	20	300	2	2	2000	4
Obs. Spk. Value	22.7	317	2.12	2.1	2180	3.57
% Recovery	114%	103%	105%	105%	106%	89%
Pb/Zn Smelter Slag duplicate						
Unadj. Conc.	0.31	0.152	0.025	0.005	1.62	<0.01
Dilution Factor	1	20	1	1	20	20
Reported Value	0.31	3.04	0.025	0.005	32.4	<0.2
Spike Added	12	120	2	2	1200	4
Obs. Spk. Value	12.6	129	2.12	2.13	1340	3.76
% Recovery	102%	105%	105%	106%	109%	94%

Preparation Procedure: SYNTHETIC RAINWATER LEACH (SRL)

Date: 29-May-86

Sample No.	CONCENTRATION (units = mg/L) As	Ba	Cd	Cr	Pb	Ag
Ca Smelter Slag						
Unadj. Conc.	<0.05	0.769	<0.004	0.005	0.112	<0.01
Dilution Factor	1	1	1	1	1	1
Reported Value	<0.05	0.769	<0.004	0.005	0.112	<0.01
Spike Added	0.2	1.5	0.1	0.1	0.2	0.2
Obs. Spk. Value	0.172	2.37	0.096	0.105	0.258	0.172
% Recovery	86%	107%	96%	100%	73%	86%
Pb Smelter Slag						
Unadj. Conc.	<0.05	0.052	0.07	0.025	11.1	<0.01
Dilution Factor	20	20	20	1	20	20
Reported Value	<1.	1.04	1.4	0.025	222	<0.2
Spike Added	4	40	60	2	9000	4
Obs. Spk. Value	5.08	45	64.2	2.2	9900	3.68
% Recovery	127%	110%	105%	109%	108%	92%
EPA Preaward #1						
Unadj. Conc.	6.98	0.029	29.7	3.07	43.4	0.063
Dilution Factor	20	20	20	20	20	1
Reported Value	140	0.58	594	61.4	868	0.063
Spike Added	280	20	1200	120	2000	4
Obs. Spk. Value	462	30.4	1970	183	3120	3.6
% Recovery	115%	149%	115%	101%	113%	88%

APPENDIX 2

QUALITY CONTROL DATA

METALS QUALITY ASSURANCE

3050 DIGESTION PROCEDURE

VERSAR INC.

Date: 29-May-86

(units=mg/L) Batch: 939.042

		ARSENIC	BARIUM	CADMIUM
Reference Standard	Found	0.284	0.444	0.235
	True	0.281	0.460	0.244
	% Recovery	101%	97%	96%
CALB. BLK.	Results	<0.05	<0.003	<0.004
REG. BLK.1	Results	<0.05	<0.003	<0.004
REG. BLK.2	Results			
Check Standard	Found	0.309	0.4	0.258
	True	0.281	0.460	0.244
	% Recovery	110%	95%	106%
SPIKE 1 Field #: Pb/Zn Slag (mg/kg)	Samp. value	288	3060	26
	Spike value	414	5040	42
	Spike added	100	2000	10
	% Recovery	126%	99%	160%

(units=mg/L)

		CHROMIUM	LEAD	SILVER
Reference Standard	Found	0.298	0.492	0.048
	True	0.313	0.488	0.052
	% Recovery	95%	101%	92%
CALB. BLK.	Results	<0.004	<0.05	<0.01
REG. BLK.1	Results	<0.004	<0.05	<0.01
REG. BLK.2	Results			
Check Standard	Found	0.31	0.50	0.048
	True	0.313	0.488	0.052
	% Recovery	99%	102%	92%
SPIKE 1 Field #: Pb/Zn Slag (mg/kg)	Samp. value	<8	17600	<20
	Spike value	10	24600	<20
	Spike added	10	7000	10
	% Recovery	100%	100%	NC

Comments: * See page 2 for duplicate results
NC = not calcuable

METALS QUALITY ASSURANCE

3050 DIGESTION PROCEDURE (page 2)

VERSAR INC. DUPLICATE PRECISION FORM

Date: 29-May-86

Batch: 939.042

(units=mg/kg)

		ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	SILVER
DUPLICATE 1							
Field #:	Samp. value	<50	810	<4	109	144	<10
Sn Slag	Dup. value	<50	718	<4	119	375	<10
	RPD	NC	-12%	NC	9%	89%	NC
DUPLICATE 2							
Field #:	Samp. value	288	3060	26	<8	17600	<20
Pb/Zn Slag	Dup. value	316	2980	28	<0.4	15300	<1.
	RPD	9%	-3%	7%	NC	-14%	NC
DUPLICATE 3							
Field #:	Samp. value	<5.	5.1	<0.4	4.2	<5	<1.
Cu Slag	Dup. value	<50.	6.5	<8	7.3	<100	<20.
	RPD	NC	24%	NC	54%	NC	NC
DUPLICATE 4							
Field #:	Samp. value	<50	190	70	36	31400	<20
Pb Slag	Dup. value	<50	196	82	40	33800	<20
	RPD	NC	3%	16%	11%	7%	NC
DUPLICATE 5							
Field #:	Samp. value	13700	13200	26400	11000	113200	83
EPA PREAWARD	Dup. value	14400	14000	27600	11900	117200	84
#1	RPD	5%	6%	4%	8%	3%	1%

Comments: NC-Not calculated due to values below detection limit

METALS QUALITY ASSURANCE

TOTAL DIGESTION PROCEDURE

V E SAR INC.

Date: 29-May-86

(units=mg/L) Batch: 939.042

		ARSENIC	BARIUM	CADMIUM
Reference Standard	Found	0.287	0.438	0.250
	True	0.281	0.460	0.244
	% Recovery	102%	95%	102%
CALB. BLK.	Results	<0.05	<0.003	<0.004
REG. BLK.1	Results	<0.05	0.122	<0.004
REG. BLK.2	Results	<0.05	<0.003	<0.004
Check Standard	Found	0.298	0.438	0.235
	True	0.281	0.460	0.244
	% Recovery	106%	95%	96%
SPIKE 1 Field #: Pb/Zn Slag (mg/kg)	Samp. value	144	3440	20
	Spike value	292	7320	44
	Spike added	400	4000	20
	% Recovery	37%	97%	120%

(units=mg/L)

		CHROMIUM	LEAD	SILVER
Reference Standard	Found	0.300	0.473	0.049
	True	0.313	0.488	0.052
	% Recovery	96%	97%	94%
CALB. BLK.	Results	<0.004	<0.05	<0.01
REG. BLK.1	Results	0.005	0.057	<0.01
REG. BLK.2	Results	<0.004	<0.05	<0.01
Check Standard	Found	0.31	0.486	0.051
	True	0.313	0.488	0.052
	% Recovery	99%	100%	98%
SPIKE 1 Field #: Pb/Zn Slag (mg/kg)	Samp. value	112	18500	<40.
	Spike value	160	33400	5
	Spike added	20	14000	20
	% Recovery	240%	106%	25%

Comments: * See page 2 for duplicate results

METALS QUALITY ASSURANCE

VERSAR INC.

TOTAL DIGESTION PROCEDURE (page 2)
DUPLICATE PRECISION FORM

Date: 29-May-86
Batch: 939.042

(units=mg/kg)

		ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	SILVER
DUPLICATE 1							
Field #:	Samp. value	<200	912	1	1170	144	<40
Sn Slag	Dup. value	<200	920	1.8	1140	150	<40
	RPD	NC	1%	57%	-3%	4%	NC
DUPLICATE 2							
Field #:	Samp. value	<200	3440	20	112	18500	<40
Pb/Zn Slag	Dup. value	152	3590	24	120	20500	<40
	RPD	NA	4%	18%	7%	10%	NC
DUPLICATE 3							
Field #:	Samp. value	<200	376	3.2	276	26	<40
Cu Slag	Dup. value	<200	384	3.6	288	<10	<40
	RPD	NC	2%	12%	4%	NC	NC
DUPLICATE 4							
Field #:	Samp. value	<200	260	72	412	29000	<40
Pb Slag	Dup. value	<200	280	80	408	28500	<40
	RPD	NC	7%	11%	-1%	-2%	NC
DUPLICATE 5							
Field #:	Samp. value	12800	13200	25200	11000	92000	17.6
EPA PREAWARD	Dup. value	13600	8000	25000	10500	109200	8
#1	RPD	6%	-49%	-1%	-5%	17%	-75%

Comments: NC-Not calculated due to values below detection limit

METALS QUALITY ASSURANCE

EP TOXICITY EXTRACTION

VERSAR INC. (units=mg/L) Date: 29-May-86
Batch: 939.042

		ARSENIC	BARIUM	CADMIUM
Reference Standard	Found	0.288	0.44	0.236
	True	0.281	0.460	0.244
	% Recovery	102%	96%	97%
CALB. BLK.	Results	<0.05	<0.003	<0.004
REG. BLK.1	Results	<0.05	<0.003	<0.004
REG. BLK.2	Results			
Check Standard	Found	0.305	0.45	0.252
	True	0.281	0.460	0.244
	% Recovery	109%	98%	103%

(units=mg/L)

		CHROMIUM	LEAD	SILVER
Reference Standard	Found	0.302	0.488	0.049
	True	0.313	0.488	0.052
	% Recovery	96%	100%	94%
CALB. BLK.	Results	<0.004	<0.05	<0.01
REG. BLK.1	Results	<0.004	<0.05	<0.01
REG. BLK.2	Results			
Check Standard	Found	0.31	0.514	0.05
	True	0.313	0.488	0.052
	% Recovery	99%	105%	96%

Comments: * See page 2 for duplicate results

METALS QUALITY ASSURANCE

EP TOXICITY EXTRACTION (page 2)

VERSAR INC. DUPLICATE PRECISION FORM

Date: 29-May-86

Batch: 939.042

(units=mg/L)

		ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	SILVER
DUPLICATE 1							
Field #:	Samp. value	<0.05	0.435	0.006	0.005	0.071	<0.01
Sn Slag	Dup. value	<0.05	0.431	<0.004	<0.004	<0.05	<0.01
	RPD	NC	-1%	NC	NC	NC	NC
DUPLICATE 2							
Field #:	Samp. value	<0.05	2.84	0.035	<0.004	35.7	<0.01
Pb/Zn Slag	Dup. value	<0.05	2.67	0.037	<0.004	36.9	<0.01
	RPD	NC	-6%	6%	NC	3%	NC
DUPLICATE 3							
Field #:	Samp. value	<0.05	0.075	<0.004	<0.004	<0.05	<0.01
Cu Slag	Dup. value	<0.05	0.047	0.009	<0.004	<0.05	<0.01
	RPD	NC	-46%	NC	NC	NC	NC
DUPLICATE 4							
Field #:	Samp. value	<0.05	0.041	2.34	<0.004	352	<0.01
Pb Slag	Dup. value	<0.05	0.057	1.74	<0.004	424	<0.01
	RPD	NC	33%	-29%	NC	19%	NC
DUPLICATE 5							
Field #:	Samp. value	1.56	0.62	556	0.48	15.5	0.061
EPA PREAWARD	Dup. value	1.62	0.64	544	0.52	16.1	0.054
#1	RPD	4%	3%	-2%	8%	4%	-12%

Comments: NC-Not calculated due to values below detection limit

METALS QUALITY ASSURANCE

EP TOX TEST WITHOUT ACID

VERSAR INC. (units=mg/L) Date: 29-May-86
Batch: 939.042

		ARSENIC	BARIUM	CADMIUM
Reference Standard	Found	0.298	0.438	0.235
	True	0.281	0.460	0.244
	% Recovery	106%	95%	96%
CALB. BLK.	Results	<0.05	<0.003	<0.004
REG. BLK.1	Results	<0.05	<0.003	<0.004
REG. BLK.2	Results			
Check Standard	Found	0.293	0.443	0.242
	True	0.281	0.460	0.244
	% Recovery	104%	96%	99%
DUPLICATE 1 Field #: Sn Slag	Samp. value	<0.05	0.095	<0.004
	Dup. value	<0.05	0.112	<0.004
	RPD	NC	16%	NC

(units=mg/L)

		CHROMIUM	LEAD	SILVER
Reference Standard	Found	0.31	0.486	0.051
	True	0.313	0.488	0.052
	% Recovery	99%	100%	98%
CALB. BLK.	Results	<0.004	<0.05	<0.01
REG. BLK.1	Results	<0.004	<0.05	<0.01
REG. BLK.2	Results			
Check Standard	Found	0.316	0.497	0.052
	True	0.313	0.488	0.052
	% Recovery	101%	102%	100%
DUPLICATE 1 Field #: Sn Slag	Samp. value	0.007	<0.05	<0.01
	Dup. value	0.007	<0.05	<0.01
	RPD	0%	NC	NC

Comments: NC-RPD is not calculated when the sample or duplicate value is less than the detection limit.

METALS QUALITY ASSURANCE

ASTM EXTRACTION PROCEDURE

VERSAR INC.

(units=mg/L)

Date: 29-May-86
Batch: 939.042

		ARSENIC	BARIUM	CADMIUM
Reference Standard	Found	0.305	0.45	0.252
	True	0.281	0.460	0.244
	% Recovery	109%	98%	103%
CALB. BLK.	Results	<0.05	<0.003	<0.004
REG. BLK.1	Results	<0.05	<0.003	0.005
REG. BLK.2	Results			
Check Standard	Found	0.305	0.446	0.262
	True	0.281	0.460	0.244
	% Recovery	109%	97%	107%
DUPLICATE 1 Field #: Cu Slag	Samp. value	<0.05	0.042	<0.004
	Dup. value	<0.05	0.032	<0.004
	RPD	NC	-27%	NC

(units=mg/L)

		CHROMIUM	LEAD	SILVER
Reference Standard	Found	0.31	0.514	0.05
	True	0.313	0.488	0.052
	% Recovery	99%	105%	96%
CALB. BLK.	Results	<0.004	<0.05	<0.01
REG. BLK.1	Results	<0.004	<0.05	<0.01
REG. BLK.2	Results			
Check Standard	Found	0.322	0.533	0.05
	True	0.313	0.488	0.052
	% Recovery	103%	109%	96%
DUPLICATE 1 Field #: Cu Slag	Samp. value	<0.004	<0.05	<0.01
	Dup. value	0.006	<0.05	<0.01
	RPD	NC	NC	NC

Comments: NC-RPD is not calculated when the sample or duplicate value is less than the detection limit.

METALS QUALITY ASSURANCE

SRL EXTRACTION PROCEDURE

VERSAR INC.

Date: 29-May-86

(units=mg/L) Batch: 939.042

		ARSENIC	BARIUM	CADMIUM
Reference Standard	Found	0.296	0.437	0.24
	True	0.281	0.460	0.244
	% Recovery	105%	95%	98%
CALB. BLK.	Results	<0.02	<0.003	<0.004
REG. BLK.1	Results	<0.02	<0.003	<0.004
REG. BLK.2	Results			
Check Standard	Found	0.291	0.464	0.242
	True	0.281	0.460	0.244
	% Recovery	104%	101%	99%
DUPLICATE 1 Field #: Pb/Zn Slag				
	Samp. value	<0.5	7.56	0.02
	Dup. value	0.31	3.04	0.025
	RPD	NC	-85%	22%

(units=mg/L)

		CHROMIUM	LEAD	SILVER
Reference Standard	Found	0.305	0.483	0.052
	True	0.313	0.488	0.052
	% Recovery	97%	99%	100%
CALB. BLK.	Results	<0.004	<0.05	<0.003
REG. BLK.1	Results	<0.004	<0.05	<0.003
REG. BLK.2	Results			
Check Standard	Found	0.312	0.466	0.047
	True	0.313	0.488	0.052
	% Recovery	100%	95%	90%
DUPLICATE 1 Field #: Pb/Zn Slag				
	Samp. value	0.006	55.2	<0.06
	Dup. value	0.005	32.4	<0.06
	RPD	-18%	-52%	NC

Comments: NC-RPD is not calculated when the sample or duplicate value is less than the detection limit.